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**A comparison of methods for early prediction of anaerobic biogas potential on  
biologically treated municipal solid waste**

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## ABSTRACT

Anaerobic gas production tests, generically Biochemical Methane Potential (BMP) or Biogas Potential (BP) tests, are often used to assess biodegradability, though long duration limits their utility. This research investigated whether simple modelling approaches could provide a reliable earlier prediction of total biogas production. Data were assessed from a non-automated biogas test on a large number of both fresh and processed municipal solid waste (MSW) samples, sourced from a mechanical biological treatment (MBT) plant. Non-linear models of biogas production curves were useful in identifying a suitable test endpoint, supporting a test duration of 50 days. Biogas production at 50 days ( $B_{50}$ ) was predicted using the first 14 days of test data, using (a) linear correlation, (b) a new linearisation process, and (c) non-linear kinetic models. Prediction errors were quantified as relative root mean squared error of prediction (rRMSEP), and bias. Predictions from most models were improved by removing the initial exponential increase phase. Linear correlation gave the most precise and accurate predictions at 14 days (rRMSEP = 2.8%, bias under 0.05%) and allowed acceptable prediction (rRMSEP <10%) both at 8 days, and at 6 days using separate correlations for each sample type. Of the other predictions, the new linearisation process gave the lowest rRMSEP (10.6%) at 14 days. More complex non-linear models conferred no advantage in prediction of  $B_{50}$ . These results demonstrate that early prediction of anaerobic gas production is possible for a well-optimised test, using only basic equipment and without recourse to external data sources or complex mathematical modelling.

**KEYWORDS:** Biogas Potential; biodegradability; anaerobic; biogas; kinetic model; Mechanical biological treatment (MBT)

## 1. INTRODUCTION

Biodegradable material in municipal solid waste (MSW) sent to landfill becomes a source of biogas, containing the greenhouse gases methane and carbon dioxide. Even where waste minimisation and source separation of recyclable materials are established, residual household waste may contain a substantial proportion of biodegradable material. The need to minimise biodegradable waste in landfill is recognised in the Council of the European Union Directive 1999/31/EC (European Union, 1999). Mechanical biological treatment (MBT) can be used to stabilise waste prior to landfilling. To assess diversion of biodegradable material from landfill, not only the quantity but also the potential biogas production, or anaerobic biodegradability, of processed material is relevant. The assessment recommended by the UK Environment Agency uses the BMc test (Turrell et al., 2009), a biogas production test run to completion under methanogenic conditions.

Anaerobic biogas production tests are bioassays often referred to generically as Biochemical Methane Potential (BMP) tests (Wagland et al., 2009), though where biogas rather than methane is determined, the term Biogas Potential (BP) would be more appropriate. There are a range of test methods for specific purposes which differ in sample preparation, operational conditions and gas collection (VDI-4630, 2006; Wagland et al., 2009, BSI, 2010; Walker et al., 2010). BP tests are reliable and require only simple, widely available laboratory equipment, though automated systems have also been used. For all BP tests, and BMP tests where methane is determined to assess biodegradability or energy potential, the dynamics of gas production are similar.

A major disadvantage of BP and BMP tests is their long duration. A BMc test may exceed 100 days (Turrell et al., 2009), while comparable tests vary between 21 and

1 100 days duration (Wagland et al., 2009). This timescale does not allow timely  
2 feedback for operational issues at an MBT plant. Long test duration has been  
3 identified as problematic for similar tests assessing potential gas production for  
4 anaerobic digestion (Stromberg et al., 2015) and post-digestion stability (Banks et  
5 al., 2013).

6 To overcome the long test duration, one approach has been to demonstrate  
7 correlation with shorter tests such as aerobic respirometric tests (Barrena et al.,  
8 2009; Cossu and Raga, 2008; Godley et al., 2007; Ponsá et al., 2008) or near-  
9 infrared spectroscopy (Ward, 2016). Data from the early stages of BMP tests have  
10 also been used to predict final values (Ponsá et al., 2011a, Stromberg et al., 2015,  
11 Da Silva et al., 2018).

12 Various kinetic models have been used to describe the form of the gas production  
13 curve and estimate the final value and parameters such as lag period and maximum  
14 rate (e.g. Donoso-Bravo et al., 2011; Shahriari et al., 2012; Stromberg et al., 2015).

15 The cumulative gas curve is typically described as sigmoidal, starting with a lag  
16 period, followed by a period of rapid gas production and finally a plateau where gas  
17 production approaches an asymptote value. This basic form has been also applied to  
18 microbial growth curves (Zwietering et al., 1990) and aerobic tests (Ponsá et al.,  
19 2011b; Tosun et al., 2008). More complex models may describe the curve shape  
20 more accurately for instance by including terms for a lag phase or multiple rate  
21 constants.

22 A range of models are potentially applicable to description of cumulative biogas  
23 production curves (Table 1). First order equations are among the simplest and are  
24 applicable where there is a single rate-limiting step (Shahriari et al., 2012). Variants  
25 include use of variable time dependence (Stromberg et al., 2015) and multiple terms

for rapidly and slowly available substrates (Junker et al., 2016; Ponsá et al., 2011b; Tosun et al., 2008). Additional parameters may allow a closer fit to the data, however increased complexity is only justified where it leads to significant improvement to predictions (parsimony principle). Both the Gompertz equation and the Logistic model (Junker et al., 2016) are most usefully expressed using easily interpreted parameters for lag, maximum rate, and asymptote value (Zwietering et al., 1990). Other models may be considered purely empirical with the parameters having no clear physical meaning, such as the Monod (Liu, 2007) and Levi-Minzi models.

Table 1: Models used to describe cumulative gas production curves with equations and references

| Name   | Equation   | References  |
|--|--|---|
| First order (FO)                                 | $B_t = B_{\infty}(1 - \exp(-kt)) , k>0$  | (Gioannis et al., 2009; Shahriari et al., 2012; Stromberg et al., 2015) |
| First order with modified time dependency (FOMT) | $B_t = B_{\infty}(1 - \exp(-kt^{\gamma})) , k>0$                                   | (Stromberg et al., 2015)  |
| First order-zero order (FOZO)                    | $B_t = C_r(1 - \exp(-k_1t)) + C_s(k_2t)$<br>where $0<A<100, 0<B<100, k_1>0, k_2>0$ | (Ponsá et al., 2011b; Tosun et al., 2008)                               |

|   |   |   |
|---|---|---|
| First order-first order (FOFO)            | $B_t = B_{\infty} (1 - C_r \exp(-k_1 t) - (1 - C_r) \exp(-k_2 t))$ <p>where <math>0 &lt; X &lt; 1</math>, <math>k_1 &gt; 0</math>, <math>k_2 &gt; 0</math>, <math>k_2 &lt; k_1</math></p> | (Ponsá et al., 2011b; Tosun et al., 2008)                     |
| First order variant - inverse time (FOIT) | $B_t = B_{\infty} \exp\left(\frac{-k}{t}\right)$  | See section 3.4.2   |
| Monod                                     | $B_t = B_{\infty} \left( \frac{kt}{1 + kt} \right)$   | (Junker et al., 2016; Stromberg et al., 2014),                |
| Monod quadratic (MQ)                      | $B_t = B_{\infty} \left( \frac{t^2}{t^2 + k_1 t + k_2} \right)$   | (Stromberg et al., 2015)                                      |
| Gompertz                                  | $B_t = B_{\infty} \exp(-\exp(k(\lambda - t) + 1))$ <p>Where <math>k = \frac{\mu_m \exp(1)}{M_{\infty}}</math></p>   | (Lay et al., 1999; Lay et al., 1996; Zwietering et al., 1990) |
| Modified Gompertz (GM)                    | $B_t = B_{\infty} \exp\left(-\frac{\theta_1 \exp(-k_1 t)}{k_1} - \frac{\theta_2 \exp(-k_2 t)}{k_2}\right)$  | (Stromberg et al., 2015)                                      |
| Levi-Minzi (LM)                           | $B_t = kt^m$  | (Ponsá et al., 2011b; Tosun et al., 2008)                     |
| Logistic                                  | $B_t = B_{\infty} \left( \frac{1}{(1 + \exp[k(\lambda - t) + 2])} \right)$ <p>Where <math>k = \frac{4\mu_m}{M_{\infty}}</math></p>  | (Zwietering et al., 1990)                                     |

1 Where:

2  $B_t$  = cumulative gas production at time  $t$ ,

- 1  $B_{\infty}$  = ultimate gas production at  $t = \infty$ ,
- 2  $\mu_m$  is maximum rate of gas production,
- 3  $\lambda$  is lag time in days,
- 4  $C_r, C_s$  are the rapidly and slowly degradable carbon fractions,
- 5  $\theta_1, \theta_2, k, k_1, k_2$  and  $\gamma$  are fitted constants.

6

7 A lag period is commonly observed while the microbial population adapts to the test  
8 conditions, and this is followed by an exponential growth or 'log' phase (Olofsson  
9 and Ma, 2011). The lag period has been assessed in various ways: fitting non-linear  
10 models which incorporate lag term, such as the Gompertz model (Behera et al.,  
11 2010; Boulanger et al., 2012; Lay et al., 1999), using the second derivative, and  
12 projecting back a tangent at the maximum rate to starting value (Swinnen et al.,  
13 2004). Junker et al. (2016) also refer to the lag as 10% of the total gas production  
14 when the plateau is reached, though this definition is not useful in the context of  
15 early prediction of the test endpoint. In the German fermentation test, GB<sub>21</sub>, the lag  
16 phase is defined as the period during which the rate of gas production remains under  
17 25% of the maximum rate of gas production in the first 21 days (BMU, 2001). For  
18 prediction of total gas production it may be more relevant to assess the exponential  
19 growth or log phase. This phase is unlikely to fit the simpler models that describe  
20 only the asymptote to the final value. Gioannis et al. (2009) fitted separate first order  
21 curves before and after peak gas production rate for landfill samples. For prediction  
22 of the total gas production, only the curve after the peak rate would be relevant.

23 Gas production curves typically do not have a clear endpoint and the criteria for  
24 ending BP or BMP tests are rarely reported. The BMc test is defined as running until



1 biogas production effectively ceases (Turrell et al., 2009). Similarly, other authors  
2 report that a test is complete when gas production is negligible (Gioannis et al.,  
3 2009; Ponsá et al., 2008). This introduces a degree of subjectivity in identifying a  
4 level of gas production that is considered to be negligible. Other tests use a fixed  
5 number of days; Wagland et al. (2009) reviewed published tests run for 21, 30, 45,  
6 60, 90 and 100 days. Another approach is to define the end of test as the first day on  
7 which the daily gas production is less than 1% of total gas production (Stromberg et  
8 al., 2015). A percentage of the maximum rate, or a fixed low daily rate could also be  
9 used. Since all of these approaches are arbitrary to some degree, standardised  
10 criteria would be beneficial.

11 The primary aim of this work was to identify a reliable method of predicting total  
12 biogas production values from test data recorded in the first two weeks of testing,  
13 assessed by low random errors and low bias. The test used (BMc) was a standard,  
14 non-automated BP test using widely available materials. Prediction should ideally be  
15 simple to apply without reference to extensive details about the sample or external  
16 data sources. To achieve this, it was necessary to explore the curve shape found  
17 and identify a suitable definition of the end-point of the test.

## 19 2. MATERIALS AND METHODS

### 21 2.1 Sample collection

22 Samples for BMc tests were taken between November 2013 and June 2017 from an  
23 active MBT plant. Material entering the plant was residual household waste  
24 containing between 58 and 73% organic material. In the plant, the waste was first

1 subjected to mechanical sorting, removing recyclable and non-compostable  
2 materials to produce a feedstock for the biological treatment, containing mean 74.5%  
3 organic material. Though imperfectly sorted, this may be considered to be organic  
4 fraction of municipal solid waste (OFMSW) and this term is used hereafter. The  
5 biological treatment was a six to seven week batch composting process.  
6 Temperature in the compost exhaust gas was monitored by the plant, typically  
7 exceeding 60°C by week 3 of the process and decreasing towards the final field. The  
8 quality of the final compost-like output (CLO) is the result of turning frequency,  
9 watering rate, and aeration regime, all of which varied over time through each batch  
10 and between batches.

11 Samples of compost feedstock (OFMSW, n=72) were taken after the mechanical  
12 sorting process and prior to biological treatment. Samples of CLO (n=76) were taken  
13 during discharge from the composting hall. All samples were a composite sample of  
14 at least ten increments taken over the period of a batch infeed or output. The  
15 sampling included an initial period of operation during which CLO material was not  
16 well stabilised. Sampling was therefore extended to include more stable CLO from  
17 periods of improved operation.

## 18 19 *2.2 Sample preparation and characterisation*

20 Each sample was hand fractionated and residual non-biodegradable components  
21 were quantified and removed. The remaining organic fraction was dried at 70°C for  
22 two days, then stored at 4°C until analysed. Each sample was ground to 4mm, and a  
23 subsample ground to 1mm for laboratory testing.

Dry matter (DM) was analysed according to EN 13040 (BSI, 2007). Loss on ignition (LoI) was determined at 550°C (Turrell et al., 2009) and used to calculate Volatile Solids (VS) as an estimate of organic matter content. Total organic carbon content (TOC) was analysed on a Shimadzu TOC-V elemental carbon analyser with a solid sample module. Total nitrogen content (TN) was analysed using the modified Kjeldahl method EN 13654-1 (BSI, 2001). Mean values for each sample type are shown in Table 2.

### 2.3. BMc test

Anaerobic biodegradability was measured using the BMc test (Turrell et al., 2009), optimised to the available supply of inoculum following guidance in VDI 4630 (VDI-4630, 2006). The inoculum was a mesophilic digestate from a local wastewater treatment plant. Each test batch included OFMSW and CLO samples, cellulose reference material ( $\alpha$ -cellulose, Sigma), and blanks containing inoculum and nutrients only; each in triplicate. The inoculum to substrate ratio was approximately 1:1 based on volatile solids (VS), incubation temperature was 35°C, and biogas was collected in tubes using a salt/acid barrier solution (Walker et al., 2009). Collected biogas volumes were recorded daily for at least the first 14 days, thereafter less frequently as the rate of biogas production reduced. Corrections for temperature, pressure and water vapour were calculated as indicated in Walker (2009). Biogas production for each sample and reference material replicate was corrected for biogas production from the blank inoculum in each batch. Results are expressed per sample volatile solids i.e.  $\text{L kg}^{-1}(\text{VS})$ . To validate the method, dry weight and LoI of the sample-inoculum mixture were determined at the end of selected tests and used with mean carbon content (52.6%) to assess the bulk loss of carbon by weight.

Comparison to the quantity of carbon contained in the biogas indicated that biogas recovery was greater than 80%, as recommended by VDI 4630, (VDI-4630, 2006).

#### 2.4 Data processing

Data processing was conducted using an *R* statistical environment (V. 3.3.1). A total of 484 replicates were analysed including OFMSW, CLO and cellulose reference material, with 17 replicates excluded due to gas leakage during the tests.

The term  $B_t$  is used herein to refer to cumulative biogas production at day  $t$  during a test; similarly, biogas production on specific days is indicated by subscript, e.g.  $B_{14}$  is gas production after 14 days,  $B_{\infty}$  is the maximum potential biogas production at time infinity.  $B_{14}$  was determined as the nearest recorded data point to 14.0 days since gas volumes were not always recorded at the same time each day.

Linear models were produced to calculate the relationship between  $B_{14}$  and  $B_{50}$  for all samples and for the different sample types. Predictions of  $B_{50}$  were made using (1) the single coefficients from the linear model for all samples and (2) using the different linear model coefficients for each sample type i.e. OFMSW, CLO and cellulose.

The linearisation of the data was conducted using linear modelling of  $\log(B_t)$  against  $1/t$  for each of the samples, after removal of the log phase data. This was the most effective attempt at linearisation as assessed by linear correlation coefficient ( $R^2$ ) for a subset of OFMSW and CLO samples.

The nonlinear models (Table 1) were fitted to all samples using the *nlsLM* function from the R package *minpack.lm* (V. 1.2-1) which uses the Levenberg-Marquardt fitting algorithm. Adequacy of fit was evaluated first by visual inspection of curves

and residual errors. The coefficient of determination,  $R^2$ , was calculated using the residual sums of squares between the actual and modelled values at each point along the curve (Equation 1), where values close to 1 indicate a good prediction. The rRMSE (whole curve) was calculated using Equation 2.

$$R^2 = 1 - \frac{(\sum(y_i - \hat{y}_i)^2)}{(\sum(y_i - \bar{y})^2)} \quad (1)$$

$$rRMSE = \sqrt{\frac{\sum(y_i - \hat{y}_i)^2}{n}} * 100 / \bar{y} \quad (2)$$

In Equations 1 and 2,  $y_i$  refers to measured datapoints;  $\hat{y}_i$  to corresponding calculated points, and  $\bar{y}$  to mean value of  $y_i$ .

The lag phase was estimated using models with a specific lag term (Gompertz, Logistic), and by projecting a tangent back from the point of maximum rate ( $\mu_m$ ) to zero gas production (tangent method) (Swinnen et al., 2004). To identify the end of the exponential (log) phases from each replicate curve, the first derivative of gas production against time was calculated for each replicate using the *predict.smooth.Pspline* function from the R package *pspline* (V. 1.0-17), giving the point of fastest rate.

Each model was assessed using the rRMSE of prediction (rRMSEP), expressed as a percentage using Equation 3,

$$rRMSEP = \sqrt{\frac{\sum(y_i - \hat{y}_i)^2}{n}} * 100 / \bar{y} \quad (3)$$

where  $y_i$  refers to actual  $B_{50}$ ,  $\hat{y}_i$  to predicted  $B_{50}$ , and  $\bar{y}$  to mean value of  $B_{50}$  over a group of samples. Values calculated for rRMSEP were compared between models using TukeyHSD multiple comparison. To test the time required to achieve rRMSEP of under 10%, incrementally increasing amounts of data from 2 days up to 30 days

were fitted to each model. The bias of each model was assessed as the mean of predicted  $B_{50}$  minus actual  $B_{50}$ , as a percentage of actual  $B_{50}$ .

### 3. RESULTS AND DISCUSSION

#### 3.1 Sample characterisation

Mean values of DM, Lol, TOC and TN for each sample type are shown in Table 2. Samples were typical of OFMSW and had a C:N ratio of around 25, expected to be suitable for composting. CLO samples were in general quite dry, with material deliberately dried towards the end of the composting process.

Table 2 Basic characterisation of OFMSW and CLO samples, mean values with standard deviation (in parentheses)

| Sample              | Dry Matter<br>% as<br>received | Lol<br>% DM | Total C<br>% DM | Total N<br>% DM | $B_{50}$<br>L/kg VS |
|---------------------|--------------------------------|-------------|-----------------|-----------------|---------------------|
| OFMSW<br>(n=72)     | 47.1 (4.0)                     | 72.5 (3.2)  | 38.9 (1.9)      | 1.5 (0.1)       | 483 (45)            |
| CLO (n=76)          | 71.2 (9.9)                     | 66.2 (5.0)  | 35.9 (2.7)      | 1.4 (0.1)       | 338 (51)            |
| Cellulose<br>(n=19) |                                |             |                 |                 | 697 (37)            |

#### 3.2 Modelling the full data

All models listed in Table 1 produced curves that approximate the shape of the actual cumulative gas production. Parameters for goodness of fit for each are shown in Table 3. Each model was also inspected visually using both the recorded values against fitted curves and collated residual errors, against both  $B_t$  values and time (the latter shown in Figure 1).

Table 3 Goodness of fit terms for each model; correlation coefficient  $R^2$  for all samples, and rRMSE (whole curve) across the duration of each test, for all samples and subsets for OFMSW, CLO and cellulose.

| Model      | $R^2$<br>All samples | rRMSE<br>All samples<br>(%) | rRMSE<br>OFMSW<br>(%) | rRMSE<br>CLO<br>(%) | rRMSE<br>Cellulose<br>(%) |
|------------|----------------------|-----------------------------|-----------------------|---------------------|---------------------------|
| GM         | 0.9985               | 1.76                        | 1.74                  | 1.64                | 1.66                      |
| MQ         | 0.9976               | 2.22                        | 2.03                  | 2.14                | 2.34                      |
| FOFO       | 0.9956               | 2.87                        | 2.43                  | 2.65                | 3.78                      |
| FOMT       | 0.9958               | 2.92                        | 2.74                  | 2.91                | 2.88                      |
| FOZO       | 0.9935               | 3.63                        | 2.50                  | 2.74                | 5.23                      |
| FOIT       | 0.9930               | 3.77                        | 2.78                  | 3.07                | 5.16                      |
| FO         | 0.9921               | 4.01                        | 3.01                  | 3.25                | 5.43                      |
| Gompertz   | 0.9890               | 4.74                        | 4.88                  | 5.12                | 3.32                      |
| Logistic   | 0.9828               | 5.91                        | 5.98                  | 6.38                | 4.41                      |
| Monod      | 0.9815               | 6.13                        | 4.72                  | 4.88                | 8.22                      |
| Levi-Minzi | 0.9067               | 13.78                       | 12.31                 | 12.52               | 15.45                     |

1 The modified Gompertz (GM) model had lowest rRMSE (whole curve) for both the  
2 whole dataset (1.76%) and separate groups of OFMSW, CLO and cellulose  
3 samples, and highest  $R^2$  value (0.9985). Visual inspection showed that the residual  
4 errors for this model were the most evenly spread across values of  $B_t$  and time. The  
5 greatest divergence from the data occurred in the first 20 days, though residuals  
6 were smaller than for other models over this period. The quadratic Monod (MQ) had  
7 the second lowest rRMSE (whole curve), at 2.22% across all samples, and showed a  
8 similar pattern of residuals though with larger errors throughout the test.

9 The models based on first-order curves all produced a similar pattern, with high  
10 errors in the first few days of the test and modelled  $B_t$  values tending to fall below  
11 actual values between days 5 to 20. The more complex two-part models, first order-  
12 first order (FOFO) and first order-zero order (FOZO), showed slightly lower residual  
13 errors throughout the test period and especially after about day 40 of the tests.  
14 These two models, and the first order with modified time dependency model (FOMT),  
15 gave relatively low rRMSE (whole curve) values and high  $R^2$ . However FOMT and  
16 the simplest first order model (FO) tended to slightly underestimate  $B_t$  after about  
17 day 40. All of these models produced values of rRMSE (whole curve) of about 4% or  
18 below on the full data set.

19 The Monod and Levi-Minzi (LM) models showed a similar but more extreme pattern  
20 of residuals, with LM overestimating the data after day 30. The Levi-Minzi model had  
21 the highest rRMSE (whole curve) (13.78%) and lowest  $R^2$  (0.9067) across all  
22 samples. In contrast, the Logistic and Gompertz models tended to produce values  
23 higher than the data between about 10 to 25 days and underestimate  $B_t$  later in the  
24 test.



A good fit to the whole curve does not necessarily imply a model will be useful for prediction, since the prediction may be sensitive to small deviations in the early part of the curve. Stromberg et al. (2015) note that the modified Gompertz model had previously been found to have the best fit over a large set of samples, though did not perform well as a predictive model from early data. It is however useful to consider the shape of the curve, especially the final plateau phase, to estimate an appropriate end-point for the test.

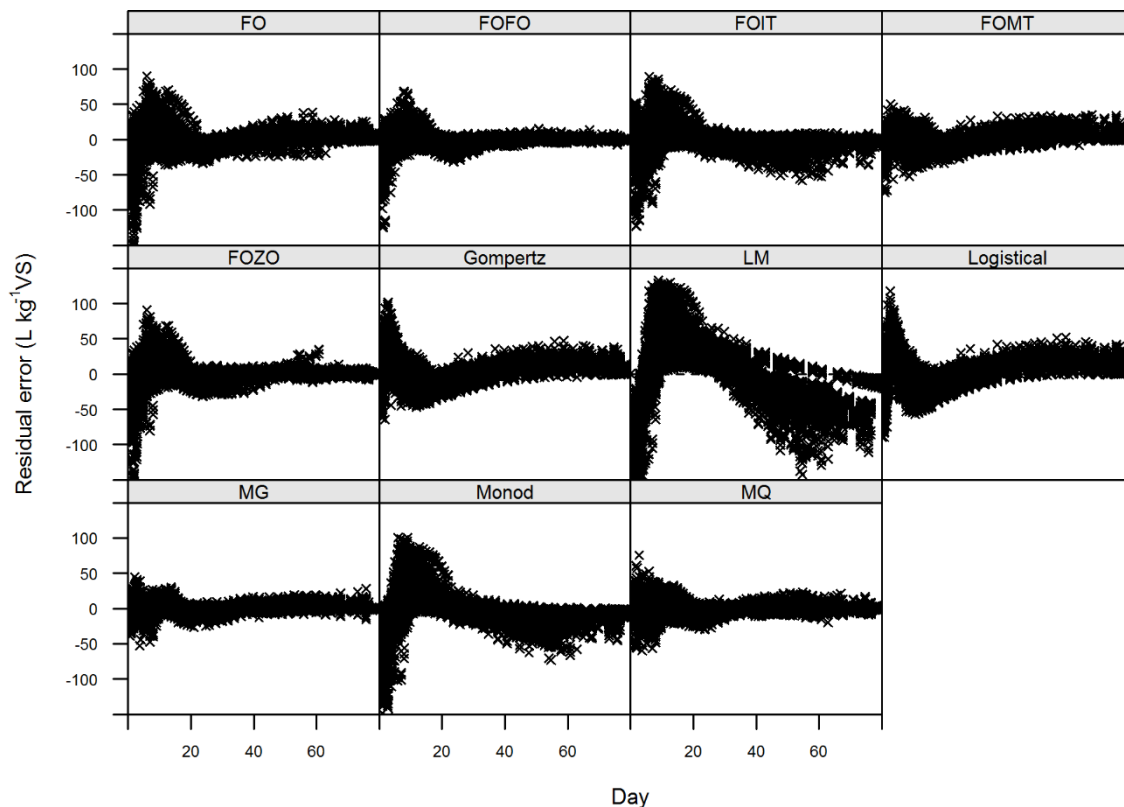


Figure 1: Residual errors between actual and modelled data over time for each non-linear model in Table 1.

### 3.3 End-point

Despite the intention for BMc tests to run until biogas production effectively ceases, there was no clear end-point and biogas production did not decrease to zero within 100 days. The cumulative curve appeared to be best described using models that tend to an asymptote at time infinity i.e. a value  $B_{\infty}$ . A reasonable definition of an endpoint would be when gas production reaches 99% of the projected endpoint i.e. the time to reach 99% of  $B_{\infty}$  ( $t_{99}$ ). It may be assumed that  $B_{\infty}$  is best estimated by models which fit the recorded data well for the final recorded values. Since the biogas production at the end of tests was small and reducing, it was assumed that the extrapolated curve for such models would remain close to the true values. Conversely, models which tend to diverge from the recorded final values would continue to overestimate (Monod, LM) or underestimate (Gompertz, Logistic) the true value of  $B_{\infty}$ . A close fit to recorded data throughout the curve is expected to give a better estimate of  $t_{99}$ .

For the simplest FO curve, the time to 99% completion was calculated as  $t_{99} = \ln(0.01)/k$ , where  $k$  is the relevant first order constant. This gave an estimate of the mean time to 99% completion of 28.6 days (maximum 46.2 days). However as noted in section 3.2, this model tends to underestimate  $B_t$  after about day 40 and is likely to give a low estimate for  $B_{\infty}$ . A slightly closer fit is achieved by FOMT, which gives a mean  $t_{99}$  of 29.0 days, with 99.4% of samples reaching the 99% target by day 50.

The two-part models FOFO and FOZO gave a close fit to the data after about day 40. For tests with the longest duration, i.e. 70 to 100 days, these two models gave the closest agreement between modelled curve and recorded data at the end of test. These attempt to identify the rapidly and slowly degradable carbon fractions. An example of each type of sample is shown graphically in Figure 2. The values found

for kinetic constants  $k_1$  (for the rapidly degradable fraction) and  $k_2$  (for the slowly degradable fraction) were comparable to those found by other authors for OFMSW in aerobic laboratory tests (Ponsá et al., 2011b; Tosun et al., 2008).

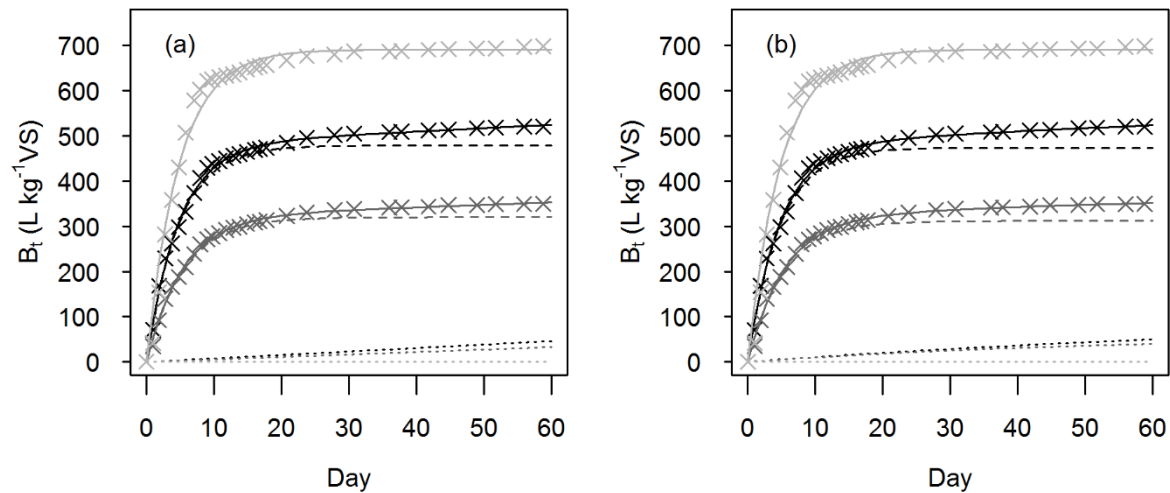


Figure 2: Graph of a) FOZO, first order-zero order model and b) FOFO first order-first order model, fit to data on typical samples of OFMSW material (black), CLO (dark grey) and cellulose (light grey), demonstrating rapidly degradable (dashed line) and slowly degradable (dotted line) fractions.

Based on the FOZO model, the slowly degradable fraction is represented as producing biogas linearly over time, with no asymptote value. If it is assumed that all carbon that is not rapidly degradable belongs to the slowly degradable fraction ( $C_s$  in Table 1, FOZO equation), the median linear rate constant  $k_2$  of  $0.00085 \text{ d}^{-1}$  corresponds to 3.2 years to completion. This may be considered unrealistically long to wait for completion of a test and slow enough to safely be ignored. This is an overestimate of  $C_s$  since there is likely to be some non-available carbon and therefore this value for  $k_2$  is an underestimate. The rate is expected to decrease

towards completion, further extending the test. If the slowly degradable fraction is ignored and the rate of rapidly degradable fraction  $k_1$  alone used to estimate the time to 99% completion, the calculated time to 99% of  $B_\infty$  is very similar to the FO model. The FOFO model failed to converge on parameter estimates for all the samples and in some cases produced simple first-order (FO) curves. However for samples that could be calculated, this model repeated the pattern found for the FOZO model. Biogas production at 50 days has been used to provide a standardised endpoint throughout this paper. This gave a robust and practical estimate of  $B_\infty$  for these tests. To verify the choice of endpoint at 50 days, values of actual  $B_{50}$  were compared to fitted values for  $B_\infty$  for other models that give the most realistic estimates for  $B_\infty$  based on the discussion in section 3.2. For the GM model, which gave the lowest rRMSE (whole curve),  $B_{50}$  was lower than modelled  $B_\infty$  by 0.58% (mean), with over 95% of the samples within 2.7% of this value. Using FO,  $B_{50}$  differs from modelled  $B_\infty$  by mean 1.6% (standard deviation 1.5%); using FOMT, mean 1.7% (standard deviation 1.3%). As expected, Monod and MQ gave higher estimates for  $B_\infty$ , and Gompertz and Logistic gave lower estimates.

This approach to establishing a fixed test duration seems appropriate for these well-optimised tests on samples from MBT. The same approach could be used in other situations provided it is possible to adequately model the curve shape. The mean rate of biogas production was 4.5 (standard deviation 1.8)  $\text{ml g}^{-1}(\text{VS}) \text{ d}^{-1}$  at 14 days, 1.6 (standard deviation 0.9)  $\text{ml g}^{-1}(\text{VS}) \text{ d}^{-1}$  at  $t_{99}$  as estimated from the FO curve, and 0.4 (standard deviation 0.4)  $\text{ml g}^{-1}(\text{VS}) \text{ d}^{-1}$  at 50 days. A gas production rate threshold could be used to specify the end of a test, for instance a threshold of 1  $\text{ml g}^{-1}(\text{VS}) \text{ d}^{-1}$ .

For comparison with the endpoint definition used by Stromberg et al. (2015), the time at which daily gas production fell below 1% of total gas volume to that point was calculated. To avoid spurious variability, the first day when 4 consecutive days were recorded below this rate was used. The mean was 17.5 days (standard deviation 3.4 days). The proportion of  $B_{\infty}$  at this point was mean 91.1% (standard deviation 3.1%).

### *3.4 Lag/log phase removal*

Cumulative curves were inspected for the length of the lag and exponential increase (log) phase. The lag phase for all these tests was found to be less than 2 days and the majority were close to zero. The effect of cutting the lag period on the rRMSE (whole curve) and  $R^2$  for each model is small. The results of other methods of lag estimation were all similar. The short lag period confirms that there was no inhibition or retarded degradation in these tests, by the definitions in VDI 4630 (VDI-4630, 2006). This may be due in part to the use of dried, ground material, which may have removed volatile inhibiting substances.

A precise definition of the end of the log phase can be made using the first derivative of the cumulative curve, or daily rate of biogas production (Figure 3). Time to the end of the log phase ranged from 0 to 6.7 days, with 96% of the samples achieving maximum daily rate of biogas production by 3 days. The mean was 1.65 days for OFMSW samples, 1.72 days for CLO samples, and 1.96 days for cellulose. Cutting the log phase gave a lower rRMSE (whole curve), and higher  $R^2$ , for the remaining data for most models. This was not true however for the Gompertz and Logistic models. These two models include a specific parameter for the lag and so may better describe the sigmoidal nature of the curves, so that removing the lag and log

phase conferred no advantage. The effect of cutting the log phase, and lag phase using the tangent method of lag removal, is shown in Figure 4.

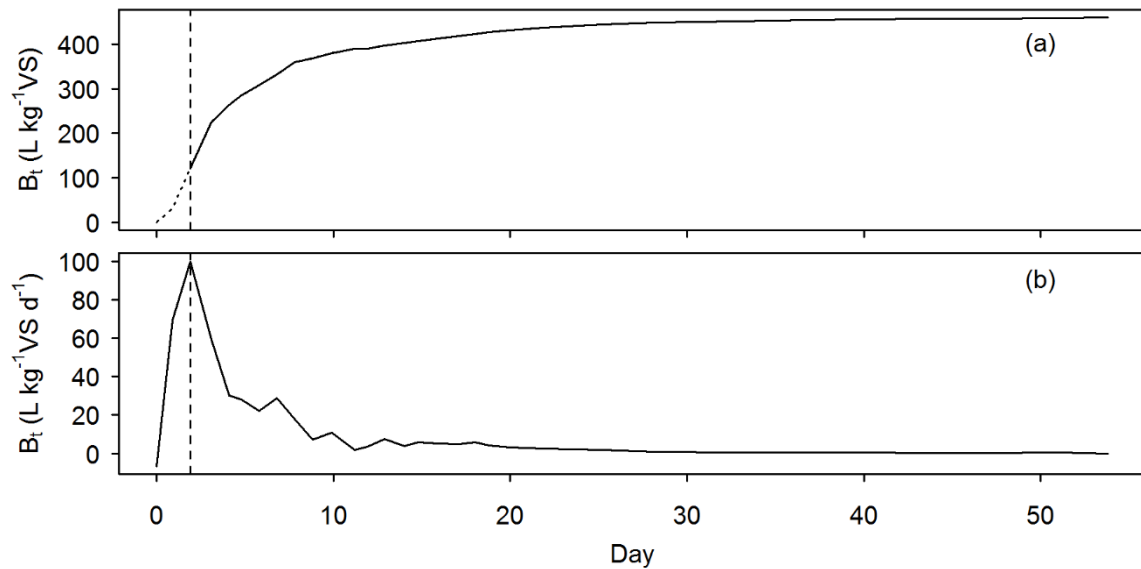


Figure 3: Example of lag identification, a) shows the cumulative curve and b) the first derivative, daily rate of biogas production. The vertical dashed line indicates the defined end of the lag period.

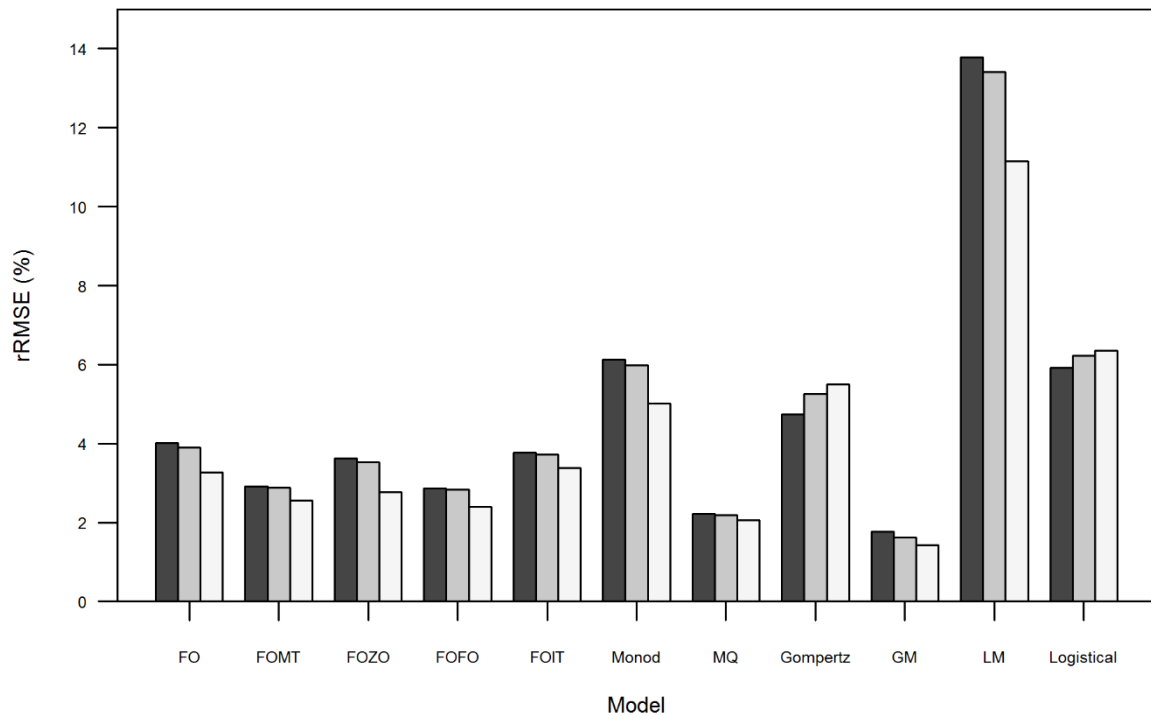


Figure 4 Error terms as rRMSE (whole curve) for a range of models with a) all data (dark grey), b) lag phase data as estimated from the tangent at maximum rate removed (light grey), c) log phase data calculated by first derivative removed (white).

Up to the end of the log phase, biogas production is assumed to be limited by the increasing population of biogas-producing microbes. After this point, biogas production is expected to be controlled by the available substrate, or more precisely, hydrolysis of the suspended reactants as the rate limiting step (Shahriari et al., 2012; Stromberg et al., 2014). This would suggest a simple first order (FO) curve would be appropriate after the log phase. This is consistent with the approach taken by Gioannis et al. (2009), who fitted separate first order curves before and after the point of maximum rate.

### 3.5 Methods of prediction of total gas production

#### 3.5.1 Simple linear correlation to 50 days

At day 14 an rRMSEP of 2.8% was achieved with the single linear model (Equation 4). No correction was made for the lag or log phase.

$$B_{50} = a + (b * B_{14}), \quad (4)$$

where  $a = 44.816$ ,  $b = 1.019$ .

It would be expected that the shape of curve will affect the relationship. In this case the BMc test is well optimised within one laboratory and it is possible that a different relationship would be found with a different physical setup or inoculum. The work by Ponsá et al. (2011a), however, supports the result, also showing good correlation between gas production from day 3 onwards to 50 and 100 days for OFMSW. Their reported correlation between 14 and 50 days produced  $R^2 = 0.939$  ( $n = 20$ ), and the equation was  $B_{50} = -24.6 + 1.49 * B_{14}$ . The higher gradient suggests their test was less advanced at 14 days. As noted in section 3.4, these tests were free of inhibition affects, which if present could change the shape of the curve and invalidate the prediction.

#### 3.5.2 Modelling by linearisation

If a cumulative curve follows a simple shape it is likely that a transformation of the data can produce a linear relationship. This allows a linear correlation to be used in a readily available program such as Microsoft Excel, without knowledge of more specialised statistical programs. Various simple transformations were tested using linear correlation ( $R^2$ ) and visual inspection of both the transformed data and plots of the resulting theoretical models as shown in Figure 5 for the most successful model.



The best linear fit was found by plotting a log of the biogas production,  $\ln(B_t)$ , against the reciprocal of time  $t$  as demonstrated in Figure 5, giving the relationship (Equation 5):

$$\ln(B_t) = a + b \left( \frac{1}{t} \right), \quad (5)$$

where  $a$  and  $b$  are the intercept and slope obtained by linear regression. This allows estimation of  $B_\infty$  from the intercept, based on a limited number of points. Removal of the early log phase data up to the maximum rate was found to improve the linear correlation and this has been used throughout.

Equation 5 can be rewritten as:

$$B_t = B_\infty \exp \left( \frac{b}{t} \right), \quad (6)$$

where  $B_t$  is the cumulative biogas production at time  $t$ ,  $B_\infty$  is the maximum BMP value and equal to  $\exp(a)$ . This relationship can also be used as a non-linear model in the same way as other non-linear models. It is referred to in Table 1 and hereafter as a first order variant, First Order Inverse Time (FOIT).

Linearising the BMP curves and estimating  $B_{50}$  with the coefficients from the linear models produces an rRMSEP of <10% by day 19. The rRMSEP at day 14 was 12.7% for the combined data, 7.8% for the OFMSW samples, 12.1% for the CLO samples and 18.5% for the cellulose.

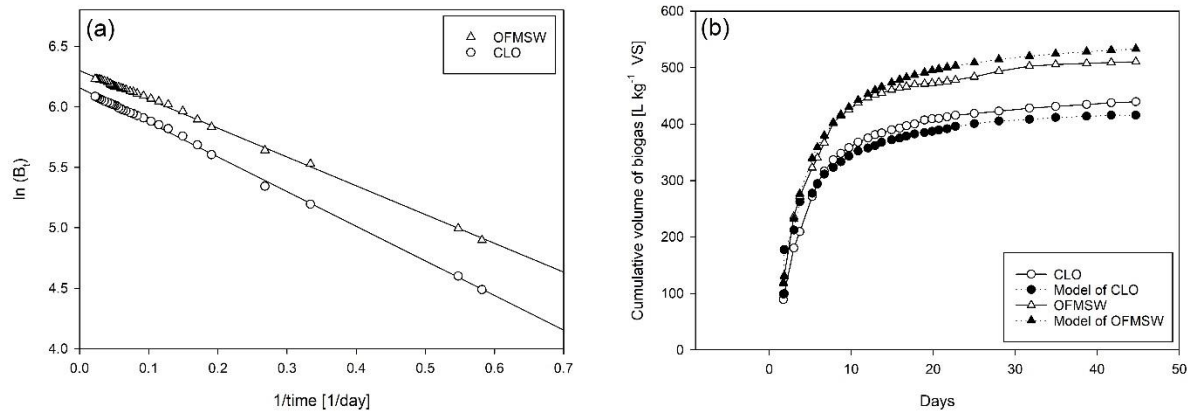


Figure 5. Typical BMc data for OFMSW and CLO sample showing (a) linearised data and (b) model prediction based on data from the first 5 days runoff the test.

### 3.5.3 Nonlinear model predictions of BMc

The non-linear model with the lowest rRMSEP for prediction of  $B_{50}$  after 14 days was the linearisation-derived FOIT model for the combined data (10.6%), the OFMSW samples (5.3%) and the CLO samples (5.8%), and the Logistic model for the cellulose (8.2%). A TukeyHSD multiple comparison of rRMSEP between models indicated no significant difference ( $p > 0.05$ ) between most models. The exceptions were LM, FOFO, and FOZO. The LM model did not describe the curve shape well and may be expected to give poor predictions. In addition, some more promising models gave poor predictions, including FOZO and MQ. The FOFO model gave a high rRMSEP of almost 61% across all the data subsets. These models have been omitted. All final predictions were made with the log phase cut from the data, which improved predictions for all models except Logistic and Gompertz.

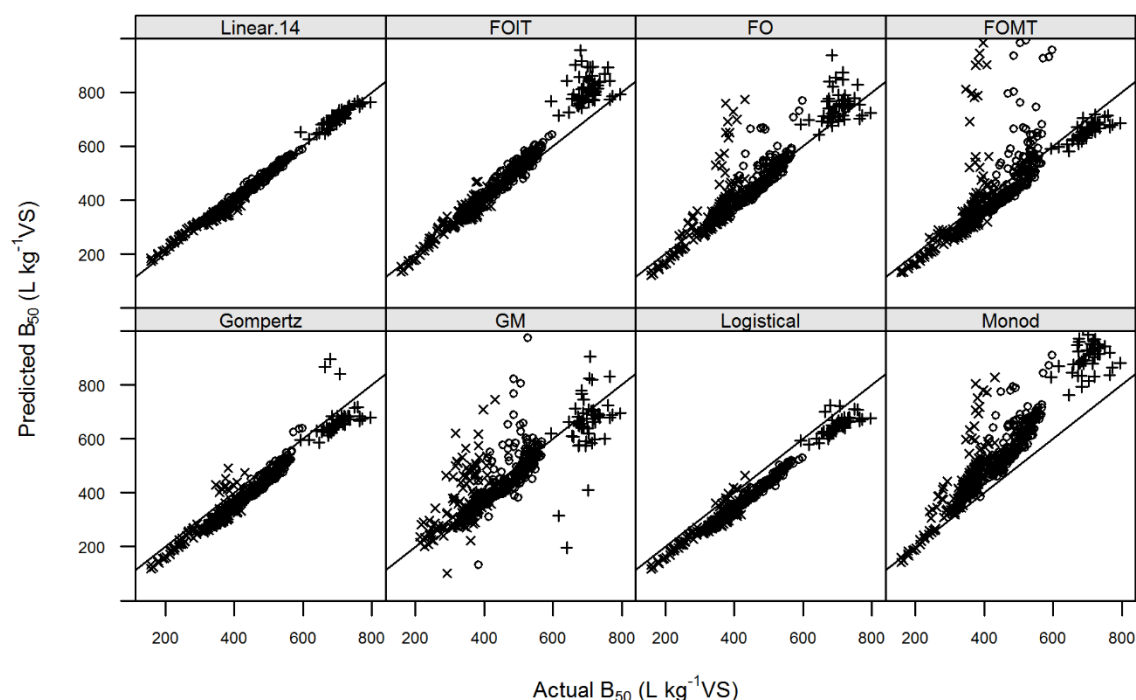
### 3.6 Comparison of predictions

Predictions of  $B_{50}$  from the initial 14 days of data, plotted against actual  $B_{50}$ , are shown in Figure 6 for linear correlation, linearisation (as FOIT model) and the more successful non-linear models. Parameters indicating variability and bias are shown in Table 4. Bias is reported separately for OFMSW and CLO samples. If the biodegradability of OFMSW and CLO is being compared, bias in opposite directions will accumulate. This is relevant for instance when calculating reduction in biodegradability across a process (Turrell et al., 2009).

Table 4 Statistical parameters for predictions of  $B_{50}$  from the first 14 days of test against actual  $B_{50}$

| Model                             | rRMSEP (%) | $R^2$ (prediction) | days to rRMSEP <10% | Bias to OFMSW samples (%) | Bias to CLO samples (%) |
|-----------------------------------|------------|--------------------|---------------------|---------------------------|-------------------------|
| Simple linear correlation         | 2.8        | 0.990              | 7.8                 | -0.34                     | -0.01                   |
| Linear correlation by sample type | 2.5        | 0.992              | 5.8                 | -0.03                     | 0.00                    |
| FOIT (linearisation)              | 10.6       | 0.857              | 15                  | 3.40                      | 1.12                    |
| FO                                | 28.6       | -0.043             | 19                  | -0.57                     | 2.75                    |
| FOMT                              | 24.8       | 0.215              | 17                  | 0.10                      | 0.65                    |
| Gompertz                          | 11.5       | 0.833              | 18                  | -9.19                     | -12.09                  |
| GM                                | 30.3       | -0.266             | 18                  | 3.25                      | 0.81                    |
| Logistic                          | 13.0       | 0.786              | 21                  | -12.15                    | -15.19                  |
| Monod                             | 39.4       | -0.979             | 28                  | 20.99                     | 24.41                   |

1



2

3 Figure 6 Predicted  $B_{50}$  from data at 14 days against actual  $B_{50}$  for each of the models  
 4 assessed. The diagonal line on each graph indicates 1:1 correspondence. OFMSW  
 5 samples (o), CLO samples (x) and cellulose (+).

6

7 The lowest bias as well as the lowest rRMSEP was achieved using simple linear  
 8 correlation. This was further improved using separate correlations by sample type  
 9 (i.e. for OFMSW, CLO and cellulose samples). Of the non-linear models, the first-  
 10 order group plus GM gave the lowest bias on OFMSW and CLO samples. Of these,  
 11 the linearisation-derived FOIT gave the lowest variability (low rRMSEP and high  $R^2$ ),  
 12 though was less successful for the cellulose reference material. This compared  
 13 favourably with predictions from respirometric activity (Scaglia et al., 2010) and with  
 14 improved predictions made by using additional parameters such as volatile solids

(Schievano et al., 2008; Schievano et al., 2009) , which achieved rRMSEP of 27.4% for the most reliable model. While it did not achieve the 10% target used by Stomberg et al. (2015) within 14 days, it does provide a simple and potentially useful prediction and can be carried out easily in a spreadsheet using linear correlation, with no requirement for additional reference data. The FO and FOMT models gave similar predictions though with a proportion of samples giving unusual high values. The GM model produced very high rRMSEP and low  $R^2$ . Predictions using the Gompertz and Logistic models were tightly clustered but showed negative bias i.e. consistently low estimates. Predictions using the Monod model were both biased and variable.

### *3.7 Earliest adequate prediction*

For each of the prediction methods in Table 4, predictions were made using test data as recorded from day 2 to day 30 of each test, in order to assess how early in the test a reasonable prediction of  $B_{50}$  could be made. A target value of rRMSEP = 10% was chosen, an arbitrary value that has also been used by other authors (Stromberg et al., 2015). It can be seen that errors increase rapidly when using less than 14 days of data for most methods (Figure 7).

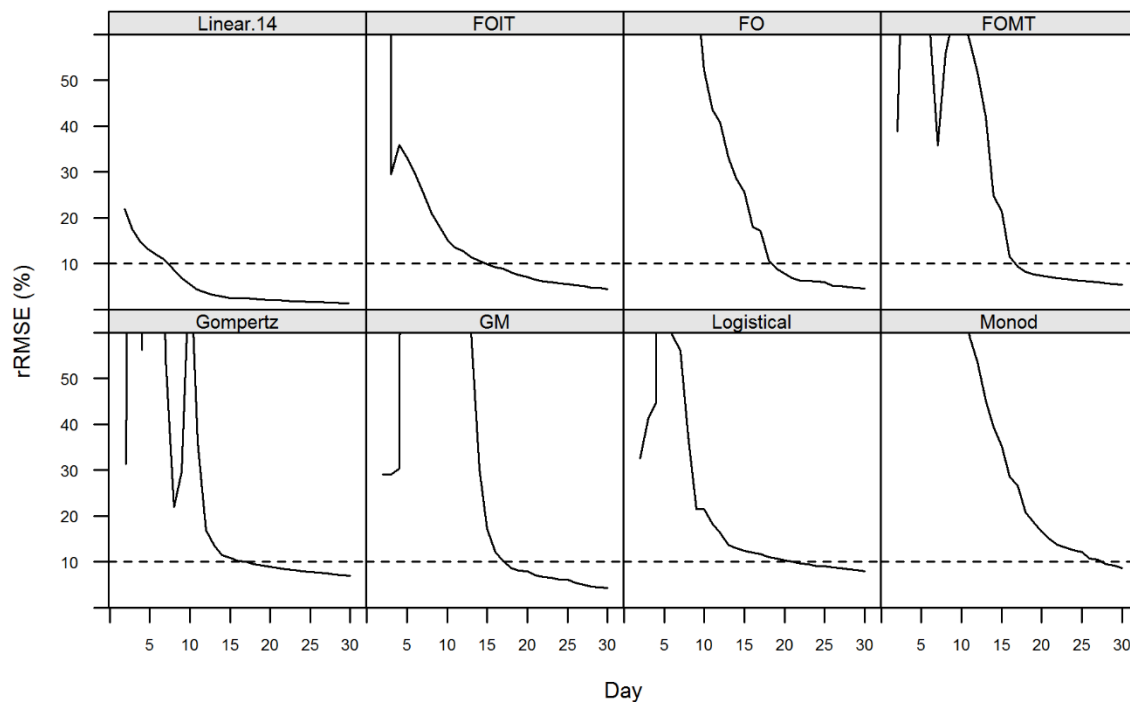


Figure 7 Error term rRMSEP for each prediction using data for increasing time periods of test. The dashed line indicates 10% rRMSEP, chosen as the threshold for adequate prediction.

Again, the most successful prediction was the simple linear correlation. Using this method, a prediction could be made earlier, achieving an rRMSEP between actual  $B_{50}$  and predicted  $B_{50}$  of  $<10\%$  for all the samples from day 8. Furthermore, an rRMSEP of  $<10\%$  could be achieved from 6 days by using multiple linear models with parameters in Equation 4 specific to sample types: for OFMSW  $a = 48.902$ ,  $b = 1.014$ ; for CLO  $a = -3.166$ ,  $b = 1.185$ ; and for cellulose samples  $a = -20.674$ ,  $b = 1.047$ .

It is possibly surprising that the simplest correlation gives the lowest error and bias as it does not account for shape of curve. However, the curve shape is in general

1 very similar in each test, with all tests run on the same optimised protocol. It appears  
2 that predictions using early data and non-linear models add more uncertainty from  
3 random variation in the data than they gain from accounting for the curve shape. In  
4 addition, bias can be increased if the curve shape changes over time, with even  
5 small changes having a strong effect on predicted values. It is known that the  
6 OFMSW samples contain a combination of slowly to rapidly available material,  
7 making such variation likely. A much more sophisticated model than those used here  
8 may overcome this, but additional complexity would be likely to lose the advantage  
9 of quick response.

10 The FO model achieved an rRMSEP of <10% after 19 days. Da Silva et al (2018)  
11 based predictions of BMP test parameters on an FO model and related the kinetic  
12 constant to predict a threshold time when final methane production and kinetic  
13 constant would be adequately independent. This indicates adequate prediction could  
14 be achieved in 13.2 days for the OFMSW with the lowest kinetic constant, and 15.3  
15 days for the CLO with the lowest kinetic constant. This is broadly consistent given  
16 the different criteria for adequate prediction. The relationship between kinetic  
17 constant and threshold time identified by Da Silva et al. (2018) explains the shorter  
18 times required to predict the maximum methane yield for more rapidly biodegradable  
19 substrates found by both Ponsá et al. (2011) and Strömberg et al. (2015).

20 Stromberg et al. (2015) achieved predictions with rRMSEP of 10% after only 6 days  
21 for household waste, using the best of a collection of models and reference to a  
22 database of known tests. As noted in section 3.3, the target endpoint of daily gas  
23 production below 1% of total used by Stromberg et al. was an earlier end-point than  
24 B<sub>50</sub> and perhaps less challenging to predict. The instruments used for this record  
25 data at equal increments of gas volume, providing more detail during the period of

rapid gas accumulation, whereas the BMc tests reported here were monitored daily. It is possible that increased data density in the early part of the test could improve predictive power. However, it is also possible that the changing shape of the curve due to biochemical changes over time are a more important limitation to prediction.

## 5. CONCLUSIONS

This study demonstrates that total biogas production in a non-automated and well optimised BMc test can be reliably predicted mathematically from data in the first 14 days of the test with reasonable variability and low bias. The most effective method was simple linear correlation which gave predictions of  $B_{50}$  ( $rRMSEP < 10\%$ , absolute bias  $< 0.02\%$ ) after only 8 days, or from 6 days using separate correlations for MBT OFMSW and CLO samples. Early reporting without recourse to additional tests can reduce costs and provide timely feedback for processing plants. These results are based primarily on MBT samples subjected to a single test methodology; further work would be required to apply these results to other sample types. However the dynamics of gas production may be expected to be similar in other tests of this type.

Gas production at 50 days was found to be a robust and practical endpoint for these tests, with over 99% of the estimated ultimate gas volume achieved for all samples. A useful alternative definition of endpoint as the point at which the rate of gas production drops below  $1 \text{ ml g}^{-1}(\text{VS}) \text{ d}^{-1}$  is suggested.

Predictions could also be made by fitting non-linear models. The most successful of these was a new model based on linearisation of the data (FOIT), which may be worth exploring further for sample types or tests where linear correlation fails. More



1 complex models, especially GM, FOZO and FOFO, most closely described the  
2 shape of the whole cumulative gas curve and provided useful insights, but conferred  
3 no advantage in early prediction of total gas production, Simpler models such as  
4 FOIT and FO were improved by first removing the initial exponential growth (log)  
5 phase.

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